FORM PTO-1390 U S DEPARTMENT OF COMMERCE PATENT AND TRADEMARK OFFICE		ATTORNEY'S DOCKET NUMBER
TRANSMITTAL LETTER TO THE UNITED STATES DESIGNATED/ELECTED OFFICE (DO/EO/US) CONCERNING A FILING UNDER 35 U.S.C. 371		4197-114
		U S APPLICATION NO (If known, see 37 CFR 1 5) 10/088751
INTERNATIONAL APPLICATION NO	INTERNATIONAL FILING DATE	PRIORITY DATE CLAIMED
PCT/DE00/03408	29 September 2000	07 October 1999

TITLE OF INVENTION
METHOD FOR PRODUCING CELLULOSE SHAPED-BODIES

APPLICANT(S) FOR DO/EO/US
BAUER, Ralf-Uwe and KIND, Uwe

Applicant herewith submits to the United States Designated/Elected Office (DO/EO/US) the following items and other information.

1. ☒ This is a **FIRST** submission of items concerning a filing under 35 U S C 371.
2. ☐ This is a **SECOND** or **SUBSEQUENT** submission of items concerning a filing under 35 U.S C 371.
3. ☐ This express request to begin national examination procedures (35 U.S.C. 371(f)) at any time rather than delay examination until the expiration of the applicable time limit set in 35 U.S.C. 371(b) and PCT Articles 22 and 39(1).
4. ☒ A proper Demand for International Preliminary Examination was made by the 19th month from the earliest claimed priority date.

5. ☒ A copy of the International Application as filed (35 U S C. 371(c)(2))
   - a ☒ is transmitted herewith (required only if not transmitted by the International Bureau).
   - b. ☒ has been transmitted by the International Bureau.
   - c ☐ is not required, as the application was filed in the United States Receiving Office (RO/US).

6. ☒ A translation of the International Application into English (35 U.S C. 371(c)(2))

7. ☒ Amendments to the claims of the International Application under PCT Article 19 (35 U.S C. 371(c)(3))
   - a. ☐ are transmitted herewith (required only if not transmitted by the International Bureau)
   - b ☐ have been transmitted by the International Bureau.
   - c ☐ have not been made; however, the time limit for making such amendments has NOT expired
   - d. ☒ have not been made and will not be made.

8. ☐ A translation of the amendments to the claims under PCT Article 19 (35 U S C. 371(c)(3))

9. ☒ An oath or declaration of the inventor(s) (35 U S C. 371(c)(4)).***(Unsigned)**

10. ☐ A translation of the annexes to the International Preliminary Examination Report under PCT Article 36 (35 U.S.C. 371(c)(5)).

Items 11. to 16. below concern other document(s) or information included:

11. ☐ An Information Disclosure Statement under 37 CFR 1.97 and 1.98.

12. ☐ An assignment document for recording. A separate cover sheet in compliance with 37 CFR 3.28 and 3.31 is included

13. ☒ A FIRST preliminary amendment
    ☐ A SECOND or SUBSEQUENT preliminary amendment.

14. ☐ A substitute specification

15. ☒ A small entity statement.

16. ☐ Other items or information

NOTE: This application is being filed with an unsigned Oath or Declaration under the provisions of 37 CFR § 1.53 in order that applicants may secure a filing date of March 20, 2002. Upon receipt of a "Notice to File Missing Parts - Filing Date Granted," an executed Declaration and Power of Attorney, will be filed in the Patent and Trademark Office. The undersigned agent affirmatively states that she has been duly authorized and appointed to file this application on behalf of the applicants and that the Declaration and Power of Attorney to be filed hereafter will confirm the undersigned agent's authorization and appointment. Applicants are entitled to small entity status within the meaning of 37 CFR § 1.9.

[redacted]

17. ☒ The following fees are submitted.	CALCULATIONS	PTO USE ONLY
Basic National Fee (37 CFR 1.492(a)(1)-(5)) Search Report has been prepared by the EPO or JPO. $860.00	JC13 Rec'd PCT/PTO 20 MAR 2002	
International preliminary examination fee paid to USPTO (37 CFR 1.482) $0 00		
No International preliminary examination fee paid to USPTO (37 CFR 1.482) but international search fee paid to USPTO (37 CFR 1.445(a)(2)). $0.00		
Neither international preliminary examination fee (37 CFR 1 482) nor international search fee (37 CFR 1 445(a)(2)) paid to USPTO..... $1000.00		
International preliminary examination fee paid to USPTO (37 CFR 1.482) and all claims satisfied provisions of PCT Article 33(2)-(4) $0.00		
ENTER APPROPRIATE BASIC FEE AMOUNT =	$ 860.00	
Surcharge of **$130.00** for furnishing the oath or declaration later than ☐ 20 ☐ 30 months from the earliest claimed priority date (37 CFR 1 492(e)).	$	

Claims	Number Filed	Number Extra	Rate		
Total Claims	17- -20 =	0	X **$18.00**	$	
Independent Claims	3- 3 =	0	X **$80.00**	$	
Multiple dependent claim(s) (if applicable)			+ **$270.00**	$	
TOTAL OF ABOVE CALCULATIONS =				860.00	
Reduction by 1/2 for filing by small entity, if applicable. Verified Small Entity statement must also be filed (Note 37 CFR 1 9, 1.27, 1.28).				$ 430.00	
SUBTOTAL =				$ 430.00	
Processing fee of **$130.00** for furnishing the English translation later than ☐ 20 ☐ 30 Months from the earliest claimed priority date (37 CFR 1.492(f)) +				$	
TOTAL NATIONAL FEE =				$ 430.00	
Fee for recording the enclosed assignment (37 CFR 1 21(h)). The assignment must be accompanied by an appropriate cover sheet (37 CFR 3.28, 3.31). **$40.00** per property +				$	
TOTAL FEE ENCLOSED =				$ 430.00	
				Amount to be: refunded	$
				Charged	$

a. ☒ A check in the amount of $430.00 to cover the above fees is enclosed.

b. ☐ Please charge my Deposit Account No. _____ in the amount of $ _____ to cover the above fees. A duplicate copy of this sheet is enclosed.

c. ☒ The Commissioner is hereby authorized to charge any additional fees which may be required, or credit any overpayment to Deposit Account No. 08-3284. A duplicate copy of this sheet is enclosed.

NOTE: Where an appropriate time limit under 37 CFR 1.494 or 1.495 has not yet been met, a petition to revive (37 CFR 1.127(a) or (b)) must be filed and granted to restore the application to pending status.

SEND ALL CORRESPONDENCE TO:

Steven J. Hultquist
Intellectual Property/Technology Law
P. O. Box 14329
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MARIANNE FUIERER
Registration No. 39,983

23448
PATENT & TRADEMARK OFFICE

4197-114
PATENT APPLICATION

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re Application of:	**Bauer, et al.**
Application No.:	New U.S. National Stage Application of PCT International Application No. PCT/DE00/03408
International Filing Date:	29 September 2000
Priority Date Claimed: 720.6) 910.0)	07 October 1999 (German Appl. No. 199 48 401.5
U.S. National Phase Filing Date:	Date of mailing identified below
Title:	**METHOD FOR PRODUCING CELLULOSE SHAPED-BODIES**

EXPRESS MAIL CERTIFICATE

I hereby certify that I am mailing the attached documents to the Commissioner for Patents on the date specified, in an envelope addressed to the Commissioner for Patents, Box Patent Application, Washington, DC 20231, and Express Mailed under the provisions of 37 CFR 1 10

Lee Ann Brown
Name of Person Mailing This Document

Lee Ann Brown
Signature

March 20, 2002
Date

EV037732967US
Express Mail Label Number

SUBMISSION UNDER 35 U.S.C. §371 OF UNITED STATES PATENT APPLICATION (NATIONAL PHASE PROCEEDINGS) BASED ON INTERNATIONAL APPLICATION NO. PCT/DE00/03408 AND CLAIMING PRIORITY OF GERMAN PATENT APPLICATION NO. 199 48 401.5

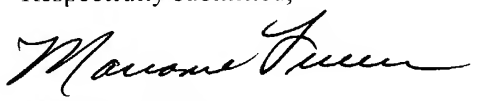
Commissioner for Patents
Box PATENT APPLICATION
Washington, DC 20231

Sir:

Submitted herewith for filing under the provisions of 37 CFR 1.53 and 35 U.S.C. § 371 is the above-referenced patent application, based on International Patent Application No. PCT/DE00/03408 and claiming priority of German Patent Application No. 199 48 401.5. A copy of the PCT International Application and related documents as originally filed are included. An English translation of the application as filed is also included. Untranslated International Preliminary Examination Report and International Search Report are included. Also included is a Preliminary Amendment, unsigned Declaration and Power of Attorney, a check in the amount of $430.00, and a transmittal letter.

Please direct correspondence relating to this application to Steven J. Hultquist, Intellectual Property Technology Law, P.O. Box 14329, Research Triangle Park, NC 27709, and direct telephonic communications relating to this application to Marianne Fuierer at (919) 419-9350.

Respectfully submitted,



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4197-114
PATENT APPLICATION

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re Application of:	**BAUER, et al.**
Application No.:	New U.S. National Stage Application of PCT International Application No. PCT/DE00/03408
International Filing Date:	29 September 2000
Priority Date Claimed:	07 October 1999 (German Appl. No. 199 48 401.5)
U.S. National Phase Filing Date:	Date of mailing identified below
Title:	**METHOD FOR PRODUCING CELLULOSE SHAPED-BODIES**

EXPRESS MAIL CERTIFICATE

I hereby certify that I am mailing the attached documents to the Commissioner for Patents on the date specified, in an envelope addressed to the Commissioner for Patents, Box Patent Application, Washington, DC 20231, and Express Mailed under the provisions of 37 CFR 1.10

Lee Ann Brown
Name of Person Mailing This Document

Lee Ann Brown
Signature

March 20, 2002
Date

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Express Mail Label Number

PRELIMINARY AMENDMENT

Commissioner for Patents
BOX PATENT APPLICATION
Washington, D.C. 20231

Sir:

Prior to examination of the above-identified new national phase patent application, please amend the application, as follows:

In the Specification [1]

Please insert on page 1, between the title of the application and the first paragraph, the following new paragraph:

CROSS-REFERENCE TO RELATED APPLICATIONS

This application is filed under the provisions of 35 U. S.C. §371 and claims the priority of International Patent Application No. PCT/DE00/03408 filed September 29, 2000, which in turn claims priority of German Patent Application No. 199 48 401.5 filed October 7, 1999.

Field of the Invention

On page 1, between the first and second paragraph, please insert the following:
BACKGROUND OF THE INVENTION

On page 2, between the first and second paragraph, please insert the following:
DESCRIPTION OF THE INVENTION

In the Claims

1. A process for manufacture of cellulose mouldings with reduced cellulose decomposition from TCF-bleached or ECF-bleached] cellulose, comprising:

dissolving a bleached cellulose pulp having a reduced number of carboxyl groups in an aqueous tertiary aminoxide to form a bleached cellulose solution, wherein the reduced

[1] Applicants has provided a marked-up version of amended paragraphs, and claims 1-8 in Appendix A, and a clean set of all pending claims, amended to date, in Appendix B.

number of carboxyl groups causes reduced decomposition of the bleached cellulose in the process, and wherein the bleached cellulose is selected from the group consisting of: TCF-bleached cellulose comprising a carboxyl group content in the range from 1 to 35 *u*mol/g and ECF-bleached cellulose comprising a carboxyl group content in the range from 1 to 50 *u*mol/g;

deforming the cellulose solution; and

coagulating the deformed solution to generate a spinning solution for forming the cellulose moulding.

2. The process according to claim 1, wherein the TCF-bleached cellulose comprises a carboxyl group content in the range from 15 to 30umol/g.

3. The process according to claim 1, wherein the ECF-bleached cellulose comprises a carboxyl group content in the range from 25 to 35 umol/g.

4. The process according to claim 1, wherein of N-methylmorpholin-N-oxide is used as the tertiary aminoxide.

5. The process according to claim 1, further comprising introducing into the bleached cellulose solution at least one organic compound comprising at least four carbon atoms, at least two conjugated double bonds, and at least two substitutes -X-H, whereby X has the significance of O or NR, and R is hydrogen or an alkyl group with 1 to 4 carbon atoms.

6. The process according to claim 1 wherein the decomposition of the cellulose is restricted to a fraction in the range from 3 to 20% by weight, related to the cellulose pulp used.

7. Use of a TCF-bleach cellulose having a carboxyl group content in the range from 1 to 35*u*mol/g for forming a cellulose solution in a solvent containing a tertiary aminoxide for the manufacture of mouldings.

8. Use of a ECF-bleached cellulose having a carboxyl group content in the range of 1 to 50*u*mol/g for forming a cellulose solution in a solvent containing a tertiary aminoxide for the manufacture of mouldings.

Please add the following new claims 9-17.

9. The process according to claim 1, wherein the cellulose moulding includes a member selected from the group consisting of: fibres, filaments, and films.

10. The process according to claim 1, wherein the cellulose moulding formed from the bleached cellulose pulp having a lower content of carboxyl groups has a higher degree of whiteness relative to cellulose pulp having a higher content of carboxyl groups.

11. The process according to claim 1, wherein the decomposition of the cellulose is restricted to a fraction in the range from 8 to 15% by weight, related to the cellulose pulp used.

12. The process according to claim 1, wherein the cellulose solution comprises about 13% cellulose, about 10.5% water and about 76.5% of NMMO.

13. The process according to claim 1, wherein the cellulose moulding formed from the bleached cellulose pulp having a lower content of carboxyl groups has a higher degree of polymerization relative to bleached cellulose pulp having a higher content of carboxyl groups.

14. The process according to claim 1, wherein the bleached cellulose pulp having a lower content of carboxyl groups has a lower degree of decomposition relative to bleached cellulose pulp having a higher content of carboxyl groups.

15. A cellulose moulding article formed from a TCF-bleached cellulose according to claim 7.

16. A cellulose moulding article formed from a TCF-bleached cellulose according to claim 8.

17. The process according to claim 5, wherein the organic compound is isopropyl gallate.

REMARKS

It is requested that the examination and prosecution of this application proceed on the basis of these amended and new claims 1-17.

Respectfully submitted,

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APPENDIX A

In the Specification

Please insert on page 1, between the title of the application and the first paragraph, the following new paragraph:

CROSS-REFERENCE TO RELATED APPLICATIONS

This application is filed under the provisions of 35 U. S.C. §371 and claims the priority of International Patent Application No. PCT/DE00/03408 filed September 29, 2000, which in turn claims priority of German Patent Application No. 199 48 401.5 filed October 7, 1999.

Field of the Invention

On page 1, between the first and second paragraph, please insert the following:
--BACKGROUND OF THE INVENTION--

On page 2, between the first and second paragraph, please insert the following:
--DESCRIPTION OF THE INVENTION--

In the Claims

Please amend claims 1 - 8 to read as follows:

1. A process [Process] for manufacture of cellulose mouldings [, such as fibres, filaments, or films] with reduced cellulose decomposition from TCF-bleached or ECF-bleached] cellulose, comprising:

dissolving [in which] a bleached cellulose pulp having a reduced number of carboxyl groups [is dissolved] in an aqueous tertiary aminoxide to form a bleached cellulose solution,

wherein the reduced number of carboxyl groups causes reduced decomposition of the bleached cellulose in the process, and wherein the bleached cellulose is selected from the group consisting of: TCF-bleached cellulose comprising a carboxyl group content in the range from 1 to 35 *u*mol/g and ECF-bleached cellulose comprising a carboxyl group content in the range from 1 to 50 *u*mol/g;

deforming [to form a mouldable cellulose,] the cellulose solution [deforms,]; and

[and the moulding comes into being by coagulation] coagulating the deformed solution to [form] generate a spinning solution for forming the cellulose moulding. [, characterized in that, to reduce the cellulose decomposition in the process, TCF-bleached cellulose is used comprising carboxyl group content in the range from 1 to 35 *u*mol/g or an ECF-bleached cellulose with carboxyl group content in the range from 1 to 50 *u*mol/g.]

2. The process [Process] according to claim 1, wherein the [characterized by the use of] TCF-bleached cellulose [comprising] comprises a carboxyl group content in the range from 15 to 30umo1/g.

3. The process [Process] according to claim l, wherein the [characterized by the use of a] ECF-bleached cellulose comprises [comprising] a carboxyl group content in the range from 25 to 35 umol/g.

4. The process [Process] according to claim 1, wherein [any of claims 1 to 3 characterized by the use] of N-methylmorpholin-N-oxide is used as the tertiary aminoxide.

5. The process [Process] according to claim 1, further comprising [any of claims 1 to 4, characterized by] introducing into the bleached cellulose solution at least one organic compound comprising [forming a cellulose solution with a content of alkalis or organic compounds, whereby the later contain] at least four carbon atoms, at least two conjugated double bonds, and at least two substitutes -X-H, whereby X has the significance of O or NR, and R is hydrogen or an alkyl group with 1 to 4 carbon atoms.

6. The process [Process] according to claim 1 wherein [any of claims 1 to 5, characterized in that,] the decomposition of the cellulose is restricted to a fraction in the range from 3 to 20% by weight, related to the cellulose pulp used.

7. Use of a TCF-bleach cellulose having a carboxyl group content in the range from 1 to 35*u*mol/g for forming a cellulose solution in a solvent containing a tertiary aminoxide for the manufacture of mouldings [according to the Lyocell process].

8. Use of a ECF-bleached cellulose having a carboxyl group content in the range of 1 to 50*u*mol/g for forming a cellulose solution in a solvent containing a tertiary aminoxide for the manufacture of mouldings [according to the Lyocell process].

APPENDIX B

In the Specification

Please insert on page 1, between the title of the application and the first paragraph, the following new paragraph:

CROSS-REFERENCE TO RELATED APPLICATIONS

This application is filed under the provisions of 35 U. S.C. §371 and claims the priority of International Patent Application No. PCT/DE00/03408 filed September 29, 2000, which in turn claims priority of German Patent Application No. 199 48 401.5 filed October 7, 1999.

Field of the Invention

On page 1, between the first and second paragraph, please insert the following:
BACKGROUND OF THE INVENTION

On page 2, between the first and second paragraph, please insert the following:
DESCRIPTION OF THE INVENTION

Pending Claims 1-17

1. A process for manufacture of cellulose mouldings with reduced cellulose decomposition from TCF-bleached or ECF-bleached] cellulose, comprising:

dissolving a bleached cellulose pulp having a reduced number of carboxyl groups in an aqueous tertiary aminoxide to form a bleached cellulose solution, wherein the reduced number of carboxyl groups causes reduced decomposition of the bleached cellulose in the process, and wherein the bleached cellulose is selected from the group consisting of: TCF-bleached cellulose comprising a carboxyl group content in the range from 1 to 35 *u*mol/g and

ECF-bleached cellulose comprising a carboxyl group content in the range from 1 to 50 *u*mol/g;

deforming the cellulose solution; and

coagulating the deformed solution to generate a spinning solution for forming the cellulose moulding.

2. The process according to claim 1, wherein the TCF-bleached cellulose comprises a carboxyl group content in the range from 15 to 30umol/g.

3. The process according to claim 1, wherein the ECF-bleached cellulose comprises a carboxyl group content in the range from 25 to 35 umol/g.

4. The process according to claim 1, wherein of N-methylmorpholin-N-oxide is used as the tertiary aminoxide.

5. The process according to claim 1, further comprising introducing into the bleached cellulose solution at least one organic compound comprising at least four carbon atoms, at least two conjugated double bonds, and at least two substitutes -X-H, whereby X has the significance of O or NR, and R is hydrogen or an alkyl group with 1 to 4 carbon atoms.

6. The process according to claim 1 wherein the decomposition of the cellulose is restricted to a fraction in the range from 3 to 20% by weight, related to the cellulose pulp used.

7. Use of a TCF-bleach cellulose having a carboxyl group content in the range from 1 to 35*u*mol/g for forming a cellulose solution in a solvent containing a tertiary aminoxide for the manufacture of mouldings.

8. Use of a ECF-bleached cellulose having a carboxyl group content in the range of 1 to 50*u*mol/g for forming a cellulose solution in a solvent containing a tertiary aminoxide for the manufacture of mouldings.

9. The process according to claim 1, wherein the cellulose moulding include a member selected from the group consisting of: fibres, filaments, and films.

10. The process according to claim 1, wherein the cellulose moulding formed from the bleached cellulose pulp having a lower content of carboxyl groups has a higher degree of whiteness relative to cellulose pulp having a higher content of carboxyl groups.

11. The process according to claim 1, wherein the decomposition of the cellulose is restricted to a fraction in the range from 8 to 15% by weight, related to the cellulose pulp used.

12. The process according to claim 1, wherein the cellulose solution comprises about 13% cellulose, about 10.5% water and about 76.5% of NMMO.

13. The process according to claim 1, wherein the cellulose moulding formed from the bleached cellulose pulp having a lower content of carboxyl groups has a higher degree of polymerization relative to bleached cellulose pulp having a higher content of carboxyl groups.

14. The process according to claim 1, wherein the bleached cellulose pulp having a lower content of carboxyl groups has a lower degree of decomposition relative to bleached cellulose pulp having a higher content of carboxyl groups.

15. A cellulose moulding article formed from a TCF-bleached cellulose according to claim 7.

16. A cellulose moulding article formed from a TCF-bleached cellulose according to claim 8.

17. The process according to claim 5, wherein the organic compound is isopropyl gallate.

METHOD FOR PRODUCING CELLULOSE SHAPED-BODIES

The invention relates to a process for the manufacture of cellulose mouldings, such as fibres, filaments, or films, from TCF-bleached or ECF-bleached cellulose, in which the bleached cellulose is dissolved in an aqueous tertiary aminoxide to form a mouldable cellulose solution, the cellulose solution deforms, and the moulding comes into being by coagulation of the deformed solution. The invention also relates to the use of a TCF or ECF-bleached cellulose pulp for the manufacture of cellulose mouldings.

The problems of the viscous process led to the development of new processes for the manufacture of regenerated cellulose mouldings, in which the cellulose is brought into solution without chemical modification. The fibres and filaments obtained from these solutions are grouped under the designation "Lyocell". Especially well-suited organic solvents are tertiary aminoxides, in particular N-methylmorpholin-N-oxide (NMMO). A process for the formation of a cellulose solution in aqueous NMMO is known from DE-A 44 41 468. The manufacture of cellulose mouldings from these solutions is described in EP-A 0 574 870.

In this solution the dissolved cellulose and the solvents, under the thermal conditions of a chemical decomposition, leads to discolourations of the spinning solution. Despite intensive washing of the cellulose mouldings formed, colouring substances remain in the mouldings, as a result of which the degree of whiteness is impaired. In order to reduce the decomposition referred to, a stabilizer is therefore added to the spinning solution. Suitable stabilizers are known from EP-A 0 047 929 and DD 218 104.

The principle is known from WO 97/23666 that the use of TCF-bleached cellulose pulp in the Lyocell process leads to mouldings with a higher degree of whiteness than the use of an ECF-bleached cellulose pulp. Both cellulose pulps are manufactured with similar high initial degrees of whiteness of about 90 % and high degrees of purity, in particular with regard to the lignin content. It therefore seems questionable whether the TCF-cellulose pulp in general produces better degrees of whiteness of the cellulose mouldings manufactured from this cellulose pulp than an ECF-cellulose pulp.

The objective on which the present invention is based is the creation of a process for the manufacture of cellulose mouldings with low cellulose decomposition, based on TCF or ECF-bleached cellulose pulp. The reduction in the cellulose decomposition is intended to be attained essentially without special measures in the lyocell process. Additional advantages of the invention are derived from the following description.

The degrees of whiteness of cellulose pulp and fibre of a number of different cellulose pulp provenances were determined, which were either ECF or TCF-bleached. The determination of the degree of whiteness of the cellulose pulp was carried out in accordance with DIN 53145, Part 2. The determination of the degree of whiteness of the fibres was effected in accordance with the method described in WO 97/23666, page 6. The average degree of polymerisation of the cellulose pulps according to the Cuoxam method amounted to about 550. The degrees of whiteness measured at the fibre are compiled in Table 1.

Table 1

Degree of whiteness/ cellulose pulp quality	**Manufacturing process**	**Initial degree of whiteness in cellulose pulp**	**Degree of whiteness of the spun fibre**
Cellunler F Messrs. Rayonier USA	ECF (Elementary Chloride-Free)	89,9	55,0
Temfilm Example 1 Messrs. Tembec Canada	TCF (Total Chloride-Free)	87,5	55,3
ALICELL Messrs. Western Pulp USA	ECF	88,2	58,6
MoDo MoDo Paper	TCF	93,7	58,4
Temfilm Messrs. Tembec Canada	TCF	89,2	58,6

It can be seen from Table 1 that with ECF and TCF-bleached cellulose pulps of different provenances the degree of whiteness lies in a narrow range between 87.5 and 93.7. The degree of whiteness. The degrees of whiteness of the spun fibres is likewise in a close range, between 55.0 and 58.6. A higher degree of whiteness of the fibres spun from TCF-bleached cellulose pulp was not perceptible.

Our researches have now revealed that the cellulose decomposition in the course of the Lyocell process, i.e. essentially from the formation through to the coagulation of the spinning solution, depends on the carboxyl group content of the bleached cellulose pulp used to form the spinning solution. In this situation it has been shown that the cellulose decomposition of the spun fibres is less, the lower the carboxyl group content is of the cellulose pulp

used to form the solution. It is therefore possible, according to the Lyocell process, to create cellulose fibres with reduced cellulose decomposition by making use of a TCF or ECF-bleached cellulose pulp with low carboxyl group content for the formation of the spinning solution.

The objective referred to above is therefore resolved with the process referred to in the preamble in that, in order to reduce the cellulose decomposition in the process, a TCF-bleached cellulose pulp is used with a carboxyl group content in the range from 1 to 35 µmol/g or an ECF-bleached cellulose pulp with a carboxyl group content in the range from 1 to 50 µmol/g. It has also been shown that the cellulose decomposition is the more restrained in the course of manufacture and processing of the extrusion solution, the lower the carboxyl group content is of the cellulose pulp used. In order to achieve low decomposition of the cellulose and the aminoxide in the Lyocell process, TCF and ECF-bleached cellulose pulps are used in the manufacture of the spinning solution of which the carboxyl group content lies within the range indicated. Cellulose pulps with the carboxyl group contents referred to can be manufactured by a number of different cellulose pulp manufacturers. As a result of the reduced decomposition of the spinning solution components, fewer coloured constituents are also derived, with the result that, as a secondary effect, the degree of whiteness of the moulding formed is also improved.

For preference a TCF-bleached cellulose pulp is used in the dissolving stage with a carboxyl group content in the range from 15 to 30 µmol/g or an ECF-bleached cellulose pulp with a carboxyl group content in the range from 25 to 35 µmol/g. The determination of the carboxyl group content of the celluloses which are to be used can be effected according to Döring; see K. Goetze, Chemical Fibres according to the Viscous Process, Vol. 2, 1997 Edition, p. 1079.

The tertiary aminoxide used as the solvent in the preferred process of the invention is N-methylmorpholin-N-oxide-monohydrate (NMMO-MH).

In a further embodiment of the invention, a cellulose solution with a content of alkalis or organic compounds can be formed, whereby the latter contain at least four carbon atoms, at least two conjugated double bonds, and at least two substitutes -X-H, whereby X has the significance of O or NR, and R can be hydrogen or an alkyl group with 1 to 4 carbon atoms. By means of these solution additives, the low decomposition achieved according to the invention can be reduced still further. The quantity of the organic compound can lie in the range from 0.01 to 0.5 % by weight, related to the quantity of the solvent. Suitable organic compounds are known from EP-A-0 047 929. A frequently used compound is isopropyl gallate.

By means of the process according to the invention, the decomposition of the cellulose is restricted to a fraction in the range from 3 to 20 % by weight related to the cellulose pulp used. For preference the decomposed cellulose fraction lies in the range from 8 to 15 % by weight.

The invention further relates to the use of a TCF-bleached cellulose pulp or an ECF-bleached cellulose pulp with a carboxyl group content in the range from 1 to 35 µmol/g or 1 to 50 µmol/g respectively for the formation of a cellulose solution in a solvent containing tertiary aminoxide for the manufacture of mouldings according to the Lyocell process. By means of the use of these cellulose pulps, not only is the decomposition in the course of the Lyocell process reduced, but the degree of whiteness of the mouldings formed is increased.

The invention is now explained in greater detail by the following examples.

Examples 1 to 6

Long-fibre sulphite cellulose pulp was bleached by alkaline peroxide-reinforced oxygen extracts, then bleached in a known manner with ozone and with peroxide. The bleaching methods are described, for example, in R.P. Singh, The Bleaching of Pulp, TAPPI Press, Atlanta, USA. In this situation, three different carboxyl group contents are used (Examples 1 to 5). Three further samples of the cellulose pulp were bleached with hypochlorite. The degree of polymerisation and the initial degree of whiteness of the pulps were determined in accordance with the methods referred to above, as were the carboxyl group and carbonyl group contents of the cellulose pulp. From the bleached cellulose pulps, spinning solutions with 13 % cellulose, 10.5 % water and 76.5 % NMMO are manufactured in a known manner. The solutions were spun in accordance with the dry-wet process at 95 °C with a nozzle of 65 µm hole diameter. The degree of polymerisation and the degree of whiteness of the fibres obtained were determined. The determination of the degree of polymerisation was effected in accordance with the Cuoxam method. The values obtained are indicated in Table 2.

Table 2

Example	1	2	3	4	5	6
Bleaching	TCF	TCF	TCF	ECF	ECF	ECF
Carboxyl group content µmol/g	20,2	24,9	34,8	31,1	35,1	41,2
Carboxyl group content*) µmol/g	52,1	48,2	35,6	26,1	24,5	82,2
DP cellulose pulp	540	547	560	555	566	550
DO fibre	519	505	470	485	465	440
DP decomposition %	3,9	7,7	16,1	12,6	16,8	20,0
Initial degree of whiteness	93,2	93,6	93,7	93,4	93,1	93,0

of cellulose pulp						
Degree of whiteness of fibre	68,9	65,4	58,4	62,8	58,5	56,7

*) For determination see K. Goetze, op. cit.

It can be seen from Table 2 that, as the carboxyl group content of the TCF-bleached cellulose pulp and of the ECF-bleached cellulose pulp increases, a substantial drop is to be observed in the degree of polymerisation and of the degree of whiteness of the fibre cellulose. For a given bleached cellulose pulp, the adjustment of the carboxyl group content accordingly opens up the possibility of improving the textile-physical properties of the spun fibre by way of the degree of polymerisation. Due to the reduced colour body formation in the course of the process, a slow discolouration of the spinning bath sets in, as a result of which costs advantages are derived in the regeneration of the solvent media.

Examples 7 to 10

The degree of polymerisation, the carboxyl group content, the carbonyl group content, and the initial degree of whiteness were determined from four bleached dissolving cellulose pulps. From the cellulose pulps, four spinning solutions were manufactured with 13 % cellulose, 10.5 % water, and 76.5 % NMMO. The zero shear viscosity of the spinning masses at 85 °C was measured (Haake RS 75, year of manufacture 1998). The spinning solutions were spun at 95 °C with a nozzle of 65 µm hole diameter in accordance with the usual dry-wet process. The degree of polymerisation of the celluloses was determined in the spinning solution, and the degree of whiteness of the fibres. The results are compiled in Table 3.

Table 3

Example	7	8	9	10
Provenance/parameters	**Tembec Temfilm MoDo Paper**		**Tembec TemSpr**	**Rayonier Cellunler**
Bleaching	TCF	TCF	ECF	ECF
Zero shear viscosity	6967	4588	4730	4720
DP cellulose pulp	538	510	520	510
DP spinning solution	490	455	452	450
DP decomposition [%]	9,9	10,8	13,1	11,7
Carboxyl group content [µmol/g]	20,9	24,4	36,8	28,8
Carbonyl group content [µmol/g]	51,3	48,2	24,6	24,3
Initial degree of whiteness	90,5	92,1	91,1	92,1
Degree of whiteness of fibre	62,3	61,2	58,3	58,0

The values from Table 3 also show that, as the carboxyl group content increases of the cellulose pulp used, the decomposition of the cellulose increases and the degree of whiteness of the fibre deteriorates in relation to the initial degree of whiteness.

Claims

1. Process for manufacture of cellulose mouldings, such as fibres, filaments, or films, from TCF-bleached or ECF-bleached cellulose, in which the bleached cellulose is dissolved in an aqueous tertiary aminoxide to form a mouldable cellulose, the cellulose solution deforms, and the moulding comes into being by coagulation of the deformed solution, characterized in that, to reduce the cellulose decomposition in the process, TCF-bleached cellulose is used comprising carboxyl group content in the range from 1 to 35µmol/g or an ECF-bleached cellulose with carboxyl group content in the range from 1 to 50 µmol/g.

2. Process according to claim 1, characterized by the use of TCF-bleached cellulose comprising a carboxyl group content in the range from 15 to 30µmol/g.

3. Process according to claim 1, characterized by the use of a ECF-bleached cellulose comprising a carboxyl group content in the range from 25 to 35 µmol/g.

4. Process according to any of claims 1 to 3 characterized by the use of N-methylmorpholin-N-oxide as tertiary aminoxide.

5. Process according to any of claims 1 to 4, characterized by forming a cellulose solution with a content of alkalis or organic compounds, whereby the later contain at least four carbon atoms, at least two conjugated double bonds, and at least two substitutes -X-H, whereby X has the significance of O or NR, and R is hydrogen or an alkyl group with 1 to 4 carbon atoms.

6. Process according to any of claims 1 to 5,

characterized in that, the decomposition of the cellulose is restricted to a fraction in the range from 3 to 20% by weight, related to the cellulose pulp used.

7. Use of a TCF-bleached cellulose having a carboxyl group content in the range from 1 to 35µmol/g for forming a cellulose solution in a solvent containing a tertiary aminoxide for the manufacture of mouldings according to the Lyocell process.

8. Use of a ECF-bleached cellulose having a carboxyl group content in the range of 1 to 50µmol/g for forming a cellulose solution in a solvent containing a tertiary aminoxide for the manufacture of mouldings according to the Lyocell process.

ABSTRACT

PROCESS FOR THE MANUFACTURE OF CELLULOSE MOULDINGS

Process for manufacture of cellulose mouldings, such as fibres, filaments, or films, from TCF-bleached or ECF-bleached cellulose, in which the bleached cellulose is dissolved in an aqueous tertiary aminoxide to form a mouldable cellulose, the cellulose solution deforms, and the moulding comes into being by coagulation of the deformed solution, characterized in that, to reduce the cellulose decomposition in the process, TCF-bleached cellulose is used comprising carboxyl group content in the range from 1 to 35µmol/g or an ECF-bleached cellulose with carboxyl group content in the range from 1 to 50 µmol/g. Cellulose mouldings having a reduced cellulose decomposition may be formed by said process.

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PATENT APPLICATION

DECLARATION AND POWER OF ATTORNEY FOR PATENT APPLICATION — **ATTORNEY DOCKET NO. 4197-114**

As a below named inventor, I hereby declare that:
My residence/post office address and citizenship are as stated below next to my name;
I believe I am the original, first and sole inventor (if only one name is listed below) or an original, first and joint inventor (if plural names are listed below) of the subject matter which is claimed and for which a patent is sought on the invention entitled:
METHOD FOR PRODUCING CELLULOSE SHAPED-BODIES
the specification of which is attached hereto unless the following box is checked:
(X) was filed March 20, 2002 as US Application Serial No. 10/088,751 or PCT International Application Number _____ and was amended on _____ (if applicable).
I hereby state that I have reviewed and understood the contents of the above-identified specification, including the claims, as amended by any amendment(s) referred to above. I acknowledge the duty to disclose all information which is material to patentability as defined in 37 CFR 1.56.

Foreign Application(s) and/or Claim of Foreign Priority
I hereby claim foreign priority benefits under Title 35, United States Code Section 119(a-d) or 365(b) of any foreign application(s) for patent or inventor(s) certificate, or 365(a) of any PCT international application which designated at least one country other than the United States of America, listed below and have also identified below any foreign application for patent or inventor(s) certificate having a filing date before that of the application on which priority is claimed:

COUNTRY	APPLICATION NUMBER	DATE FILED	PRIORITY CLAIMED UNDER 35 U.S.C. 119
Germany	199 48 401 5	07 October 1999	YES: X NO:
PCT	PCT/DE00/03408	29 September 2000	YES: X NO:

U.S. Priority Claim
I hereby claim the benefit under Title 35, United States Code, Section 120 of any United States application(s) listed below and, insofar as the subject matter of each of the claims of this application is not disclosed in the prior United States application in the manner provided by the first paragraph of Title 35, United States Code Section 112, I acknowledge the duty to disclose material information as defined in Title 37, Code of Federal Regulations, Section 1.56(a) which occurred between the filing date of the prior application and the national or PCT international filing date of this application:

APPLICATION SERIAL NUMBER	FILING DATE	STATUS(patented/pending/abandoned)

POWER OF ATTORNEY:
As a named inventor, I hereby appoint the following attorney(s) and/or agent(s) listed below to prosecute this application and transact all business in the Patent and Trademark Office connected therewith.

(2) **Steven J. Hultquist, Reg. No. 28021** **Marianne Fuierer, Reg. No. 39983**

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I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issued thereon.

1-00
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DECLARATION AND POWER OF ATTORNEY FOR PATENT APPLICATION (continued)

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2-00

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